

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

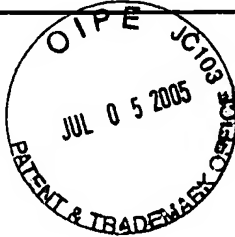
Applicant(s): Roberts et al.

Application No.: 10/673,002

Filed: 9/26/2003

Title: RESILIENT POLISHING PAD FOR
CHEMICAL MECHANICAL POLISHING

Attorney Docket No.: 02029US



Art Unit: 3723

Examiner: M. Rachuba

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF LAURENT VESIER UNDER 37 C.F.R. 1.68

Dear Sir:

I, Laurent Vesier, inventor of the subject application declare under the penalty of perjury under the laws of the United States of America that the following is true and correct.

1. The invention was conceived as early as September 18, 2002. **Appendix A1** of the Declaration shows a calendar entry for a meeting with Black Brothers on September 18, 2002 to discuss "hot melt adhesives to laminate top pads and subpads together".
2. **Appendix A2** shows notes from the above-noted September 18th meeting.
3. **Appendix B** shows a memo ("Adhesive Improvement Project") prepared on October 8, 2002 summarizing the research efforts in stacked pads, in particular, work on hot melt adhesives during at least between September 25, 2002 and October 9, 2002. For example, on page 8 of the memo, a chart illustrating shear strength tests between polishing pads having hot melt adhesives and conventional pressure sensitives adhesives is shown.

4. **Appendices C1-C4** are emails to various parties, discussing the subject invention during that time in an effort to reduce the invention to practice:

C1. Message to P. Freeman dated September 26, 2002 attaching an outline for the hot melt adhesives project.

C2. Message to co-inventor, John Roberts, dated September 26, 2002 regarding use of a particular reactive hot melt vendor.


C3. Message to other Rohm and Haas Electronic Materials CMP Inc. employees dated October 2, 2002 regarding a test procedure for shear strength testing of hot melt adhesives.

C4. Message to Rohm and Haas Company scientists dated October 3, 2002 regarding selection of hot melt adhesives.

5. **Appendix D** shows a printout of an email dated October 9, 2002 to the Rohm and Haas Electronic Materials CMP Inc., law department, attaching the invention disclosure ("Hot Melt Adhesive NOI") of the present application.

Respectfully submitted,

6/30/2005
Date



Laurent Vesier

APPENDIX A1

Meeting☐ Notify me 
☐ Mark Private ☐ Pencil In**Subject** Hot melt adhesive laminators, et al.**Chair** John Roberts/NAmerica/Rodel**When**
Starts Wed 09/18/2002 09:00 AM
Ends Wed 09/18/2002 10:00 AM 1 hour**Where**
Location
Rooms Conference Room:
4/DELAWARE**Invitees**
Required (to) Chau Duong/NAmerica/Rodel@EM, Darrell String/NAmerica/Rodel@EM, David James/NAmerica/Rodel@EM, George Feeley/NAmerica/Rodel@EM, Laurent S. Vesier/NAmerica/Rodel@EM**Categorize****Description**

Edwin Patrick from Black Brothers will be here at 9 AM on 9/18. Their expertise is in hot melt coaters and lamination systems.

This is to start the investigative process in using hot melt adhesives to laminate top pads and sub pads together, as well as directly applying hot melt PSAs to the bottom of the sub pad.

The goal is to develop a robust, high strength system for attaching pads to each other, as well as giving us control over PSA types and application. Hopefully, we will also, as a side benefit, see a reduction in the COO of these systems.

Your Notes

APPENDIX A2

↳ $ny \rightarrow PM$

- \$ 12,000

LASER

fi- n vick.

15 years.

↓ 8% NW rate in 6% AT

9-18-82 BLACK BROTHERS / MTF ~~ADTS~~
PRIMARY REACTO of ISOCYANATES

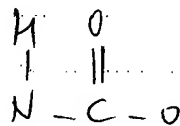
WITH ACTIVE H_2 COMPODS OCCURS EASILY w/TH HEAT ↑

H_2 NOT PRESENT IN PUFFS.

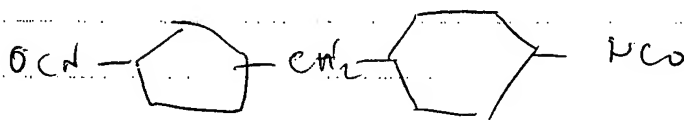
2 phase structure: hard / soft (matrix)

~~WAD STG 115~~ ~~47~~ ~~576276276~~

WALD. JZOTJ → HZLO IN DISPERSED DOMAINS VDW + H₂

$$R-OH + R'CO$$


MDI terminated; impacts moldability.
Low vapor P.



$N_{10}/0.01 \dots 1.0 \text{ R.T. } 1 \text{ } \sigma_f \uparrow$

2-12

APPENDIX B



Laurent S.
Vesier/NAmerica/Rodel
06/29/2005 02:44 PM

To Edwin Oh/NAmerica/Rodel@ROH
cc
bcc
Subject Application number 10/673002

Ed,

Attached is a memo summarizing our research efforts in stacked pads, in particular, adhesives for stacked pads during at least the critical time period that we discussed. Namely, this memo summarizes work on hot melt adhesives during at least between September 25, 2002 and October 9, 2002. For example, on page 8 of the memo, a chart illustrating shear strength tests between polishing pads having hot melt adhesives and conventional pressure sensitives adhesives is shown. As an aside, although this memo is not dated, you will note that the "last modified" date was october 8, 2002, which was the date when the memo was prepared.



Adhesive improvement project.doc

Laurent Vesier | CMP Technologies | Rohm and Haas Electronic Materials | 451 Bellevue Road | Newark, DE 19713 |

Office: 302 366 0500x6314 Cell: 302-420 5166 | Fax: 302-453 1302 | www.electronicmaterials.rohmhaas.com



SUBJECT: Adhesive improvement project

DATE:

PREFACE:

This report is a compilation of the work done to date on increasing Rodel's understanding of the phenomena involved in delaminations of the stacked pads configurations manufactured at our Newark, DE site.

I Introduction:

The objective of this project was to increase Rodel's understanding of the parameters that influence the life of our pads adhesives. Customer complaints about the degradation of the adhesives used in stacked pads have been consuming Rodel resources in the last 5 years. As the price of the pads has increased, the intensity of these resources has also steadfastly increased as data analyses, experiments were undertaken to solve these issues.

Rodel proactively engaged in this upfront understanding in order to prevent the occurrence of these problems.

II Initial data analysis:

The project was divided in two folds: - existing pads and new adhesive technologies

II.1.1 Existing pads

II.1.2 Customer complaint data

The source of the complaints was analysed with the data compiled from Sierra Vista customer complaints. The data showed that:

III.3 Design conditions: evaluation of pad interface shear stresses

The initial assumption is that this corresponds to a plane stress problem (thin and large diameter pad), ie Z is \ll pad width, Z direction strain is zero

Two types of calculations will be evaluated:

- stresses from downforce only
- stresses from pad to wafer friction.

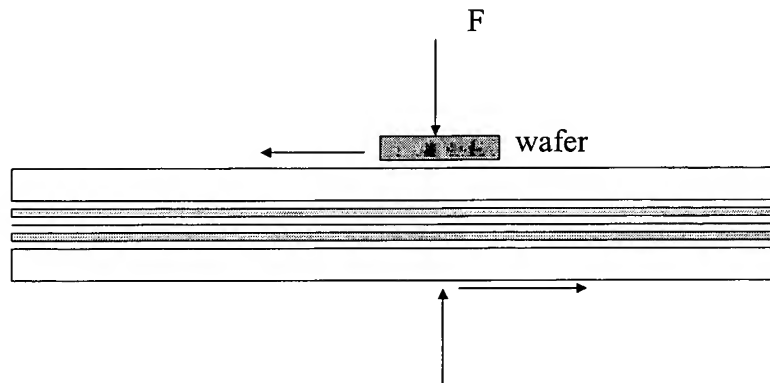
1. Evaluate stresses in X,Y directions due to downforce only:

p0	psi	14	14	14	14	14	14	14	14
a	in	4	4	4	4	4	4	4	4
x	in	4	4	0	1	2	3	4	0
y	in	0.03	0.04	0.05	0.05	0.05	0.05	0.06	0.07
txy	psi	-4.45628	-4.45623	0	-0.00079	-0.00247	-0.01089	-4.45609	0
σ_x	psi	1.583757	1.588078	2.133336	2.038378	1.955914	1.942099	1.596717	2.17287
σ_y		-1.91055	-1.99392	-2.04083	-1.98558	-0.75198	2.17287	2.103603	2.095623
$txy = -4/PI * p0 * a * x * y / ((x-z)^2 + y^2 * ((x+a)^2 + y^2))$									
shear stress from a uniform pressure distribution									

conclusion : non friction related shear due to downforce is negligible

2. Stresses from pad to wafer friction.

In the unlikely scenario that the forces are concentrated at a wafer's edge, the shear stress can be calculated as in Appendix 1.



Pad/wafer and bottom pad interfaces along with directions of forces on the interfaces

In the more likely scenario of an even stress distribution, the table below shows a simple approximation of shear stress at the interface (including thermal stress mismatch)

Downforce on wafer	14 Psi
Normal force	703.7168 Lbf
Shear stress from downforce	5.6 Psi
Shear stress from CTE mismatch	9 Psi
Shear stress from contact stresses	5
Total interface stresses	19.6 psi

Thermal stress at Suba to IC interface
CTE

IC Y's modlus

	9.20E-05 in/in/f	Polyurethane	
	6.90E-05 in/in/f	(nylon)	
E(adhesive) x epsilon from CTE		9.66 psi	87000psi E from research, IC top i stress should be lower
E(Suba) x epsilon from CTE			500MPa E top pad=
epsi from cte, assuming interface max temp of 100F, from initial 70F		0.00069	

Conclusion: shear forces are predominantly exerted on the bottom pad to top pad adhesive interface by the pad friction during polishing. Apart from unlikely stress distributions (concentration at a wafer's edge), the shear stresses are low (19psi) in comparison with reported adhesive ultimate shear stresses of epoxy and acrylic type adhesives. As the ultimate stress of the interfaces varies with strain rates, it will be difficult to test at the application conditions which vary between a minimum of 90 ipm and upper regimes of 2500-4000ipm (appendix 2). Instron tensile testers are generally designed for 0.05 to 20 ipm. Most peel tester at 90 ipm can give a lower bound indication of behavior in the "slow" polishing speeds. The influence of polishing pressure, pad surface roughness or slurry particles sizes on shear stresses are not characterized in this approximation. These variables may play a role in varying shear stresses at the pad interface. Other variables which could add to these friction related shear stresses:

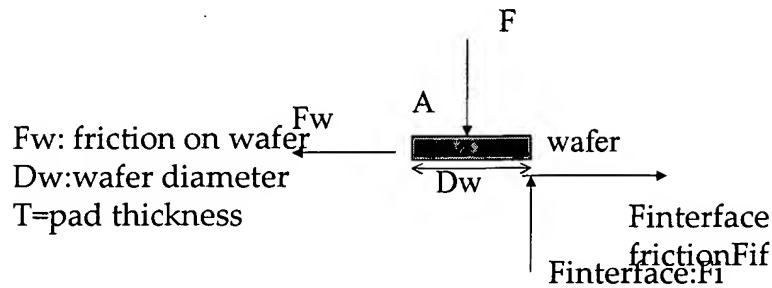
- residual stresses from top pad to psa lamination
- residual stresses from top pad/psa to suba lamination
- stresses from differential expansion mismatch between the IC and Suba pads when subpads absorb slurries

It should also be noted that the interface is really subjected to cyclic shear stresses as the wafer moves in and out of the pad areas. Even though shear stresses to rupture should be a good indicator of fatigue performance, future tests could take this damage mechanism into account. Some ASTM tests are available to test this cyclic mode of loading in adhesive interfaces.

It has been shown that the measurement of peel strength of laminates was related to the adhesive fracture toughness, G_A , (also known as adhesive fracture energy or interfacial work of fracture, and sometimes also termed G_c). (A. J. Kinloch, C. C. Lau, and J. G. Williams Int. J. Fracture, 66, (1994), pages 45-70). Therefore, peel tests will be conducted as part of this study to understand the effects of the slurry on the adhesives fracture energy. The results of these tests will be used to characterize this work of adhesion and make relative comparisons of the relative benefits of the different types of PSA.

Appendix 1:

Worst case assumption : Resulting shear stress from concentrated forces at a wafer's edge (unlikely scenario)



$$\begin{aligned} \text{Sum forces} &= 0 & \text{sum moments/A} &= 0 \\ F_{if} + F_i + F + F_w &= 0 & -FD_w/2 + F_i D_w &= 0 \\ /x: F_{if} &= F_w & \text{sum moments/B} & \\ /y & & F_{if} * t/2 + F_w &= 0 \\ & & F_{if} = -F_w * 2/t &= 0.9 * 14 * (3.14 * 8^2/4) * 2/.1 = 12000 \text{ lbs} \\ & & F_{if} = -F_w * 2/t &= 0.4 * 14 * 3.14 * 8^2/4 * 2/.1 \\ & & f: &0.4 \\ & & \text{Load:} &5629.734 \\ & & \text{shear stress on that surface area:} &112 \text{ psi} \end{aligned}$$

Friction on wafer: $F_w = f F_a = f F$
 f ; coeff of friction, F_a ; adhesion forces on wafer

$$\begin{aligned} \text{Sum forces} &= 0 & \text{sum moments/A} &= 0 \\ F_{if} + F_i + F + F_w &= 0 & -FD_w/2 + F_i D_w &= 0 \\ /x: F_{if} &= F_w & \text{sum moments/B} & \\ /y & & F_{if} * t/2 + F_w &= 0 \\ & & F_{if} = -F_w * 2/t &= 0.9 * 14 * (3.14 * 8^2/4) * 2/.1 = 12000 \text{ lbs} \\ & & F_{if} = -F_w * 2/t &= 0.4 * 14 * 3.14 * 8^2/4 * 2/.1 \\ & & \text{Coefficient of friction } f: &0.4 \\ & & \text{http://www.innovative-planarization.com/Presentatic} & \\ & & \text{Load:} &5629.734 \\ & & \text{shear stress on that surface area:} &112 \text{ psi} \end{aligned}$$

Appendix 2:

Linear velocities:

Assuming:

r =Diameter of Wafer (mm) 100

$O1$ =Platen Rotational Speed (rpm) 140

Linear Velocity (m/s) 1.46, 1460mm/s, 57in/s=3448 ipm

($v=2\pi r \omega / 60$ when ω is in rpm)

$O2$ =wafer Rotational Speed (rpm) 37

Linear Velocity (m/s) .38, 380mm/s, 14.9in/s=897 ipm

($v=2\pi r \omega / 60$ when ω is in rpm)

Maximum relative velocity: $O1-O2= 2550\text{ipm}$

r =Diameter of Wafer (mm) 100

$O1$ =Platen Rotational Speed (rpm) 40

Linear Velocity (m/s) 0.418, 418mm/s, 16.45in/s=987 ipm

($v=2\pi r \omega / 60$ when ω is in rpm)

$O2$ =wafer rotational speed 37

Linear Velocity (m/s) .38, 380mm/s, 14.9in/s=897 ipm

($v=2\pi r \omega / 60$ when ω is in rpm)

Minimum relative velocity: $O1-O2= 90\text{ ipm}$

II.1.4 Product requirements and adhesive comparison process

Product requirements were brainstormed and used in a comparative evaluation process.

Shear resistance, resistance to slurries were identified as key requirements.

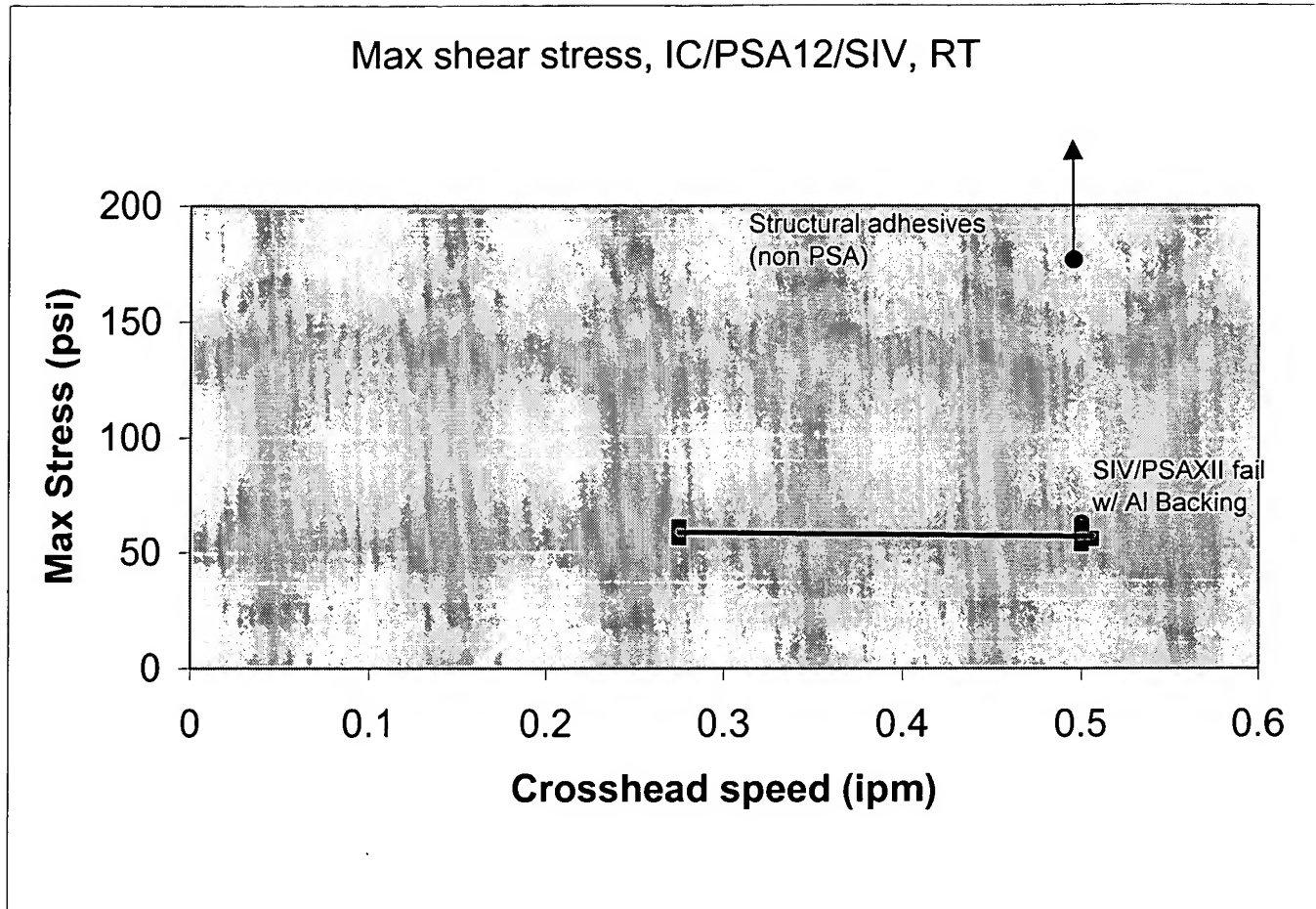


Chart above shows that the PSA used in our stacked pads have maximum shear stresses that are lower than values obtained from other technologies, ie direct coating of adhesives on IC or sub surfaces (for examples use of hot melts or other adhesives). Test followed ASTM D1002 general concepts. Test conditions: dry samples, RT, 73%RH.

II.3 End process requirements – slurries

Discussions of the types of slurries to use in determining which PSA should be used in our customers processes. Knowing that

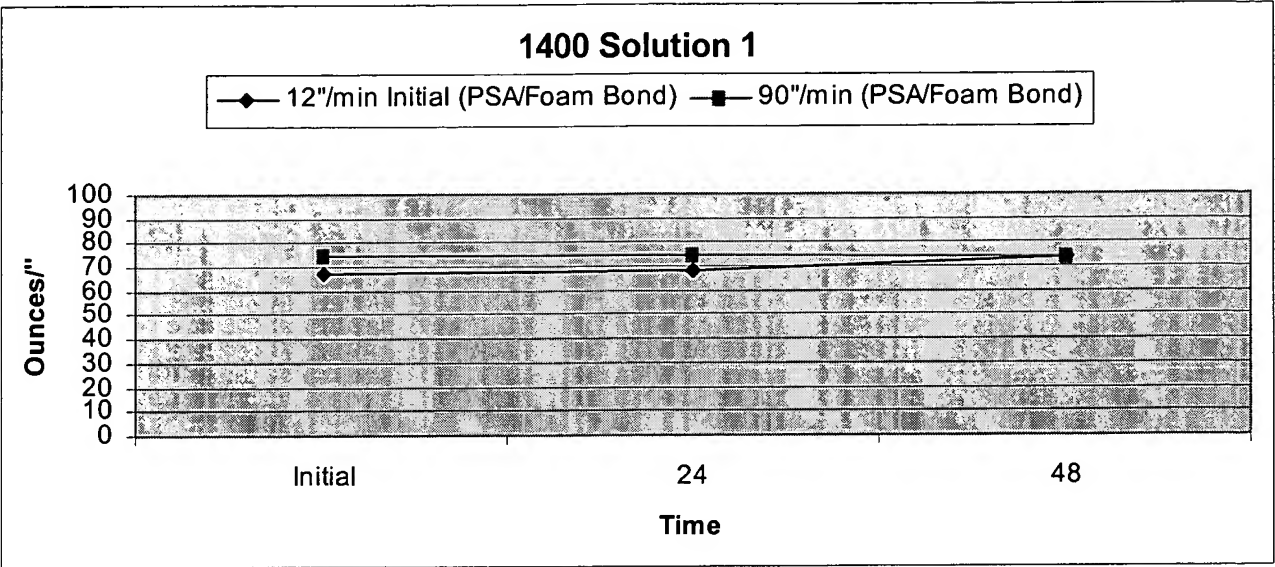
II.4 Compatibility tests data

II.4.1 Solution 1

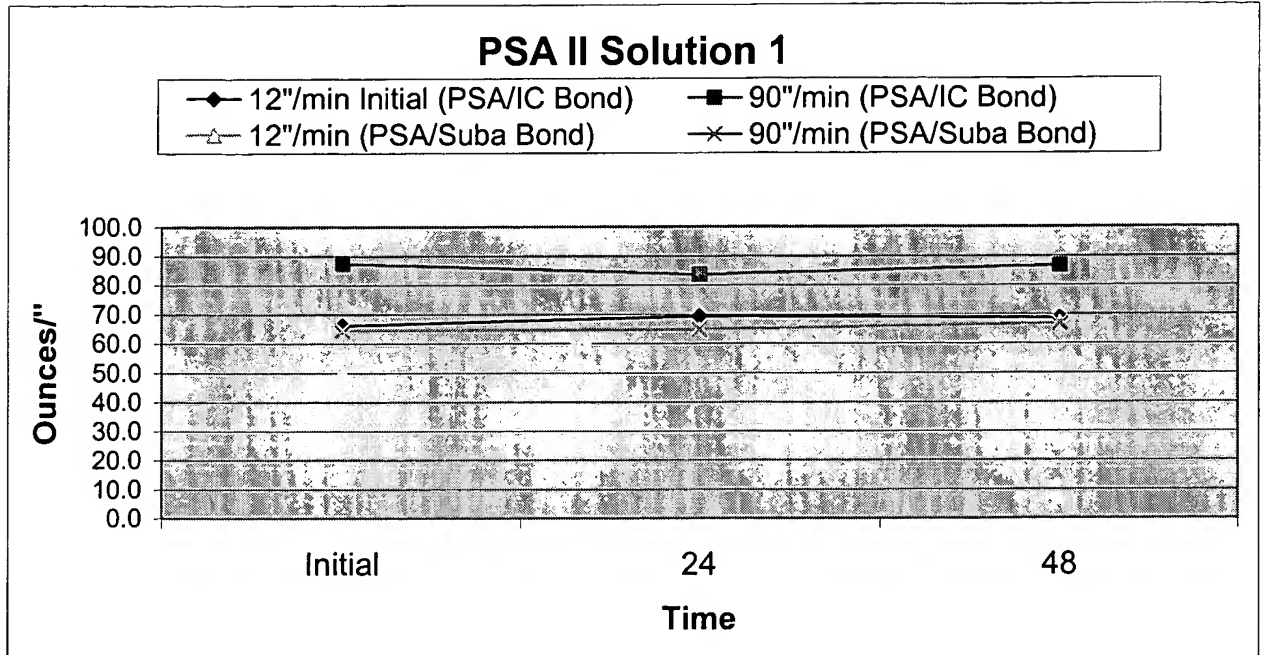
Solution 1 was composed of pH11, DIH20

II.4.1.1 Tpeel results in three test slurries

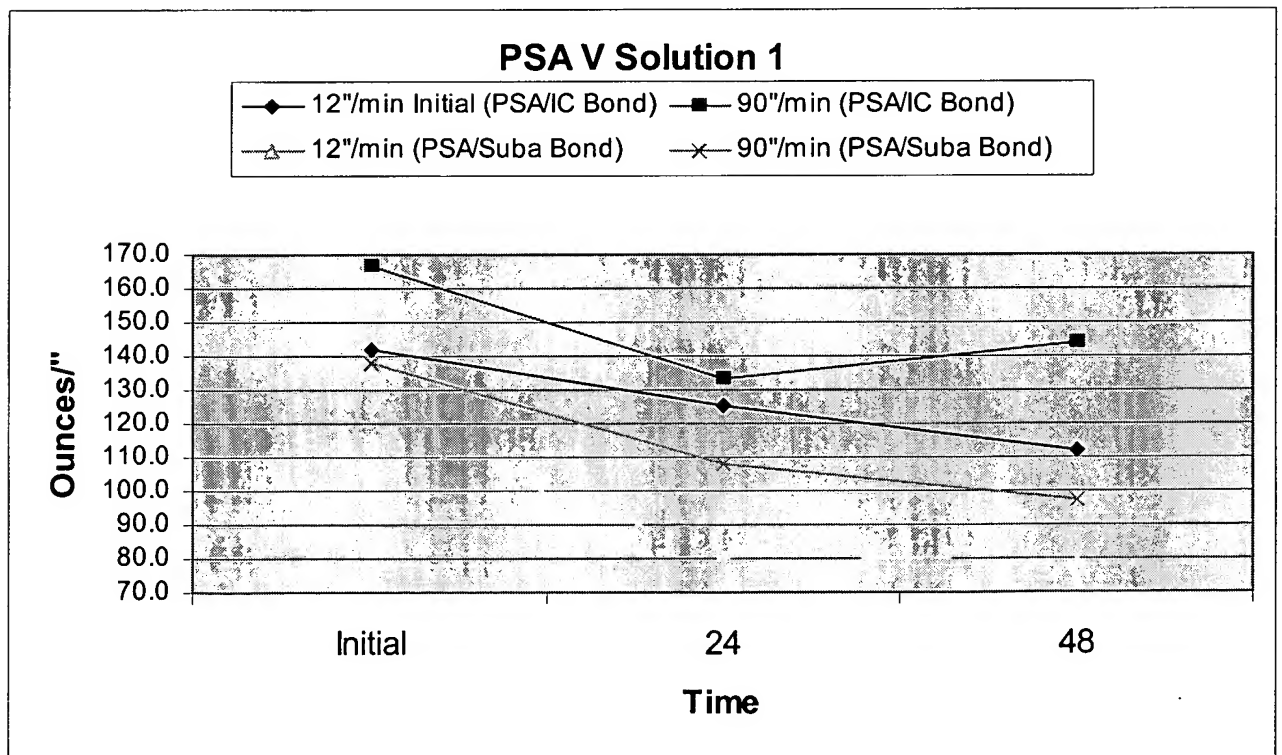
The graph below shows the 1400 foam product is not significantly affected by immersion in this caustic solution at either 12 or 90"/min



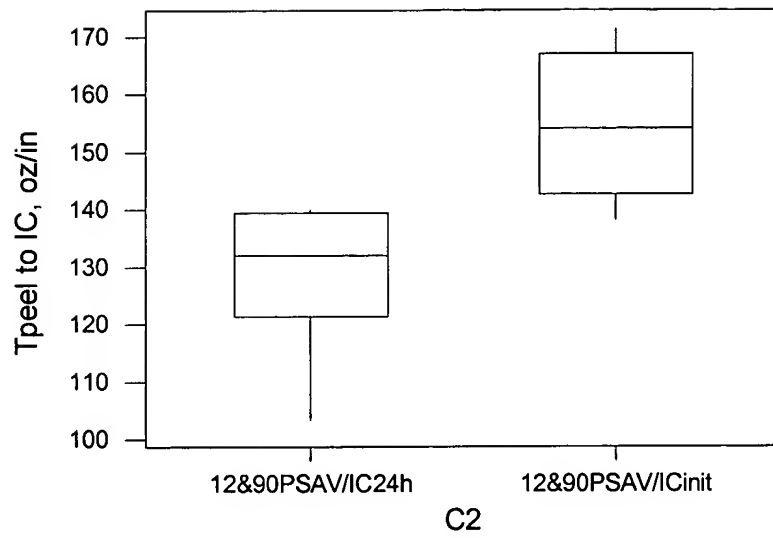
The PSAII graph below shows that this PSA is not significantly affected by the slurry immersion. The initial PSA/Suba bond lower peel strength than after immersion could be due to experimental issues.



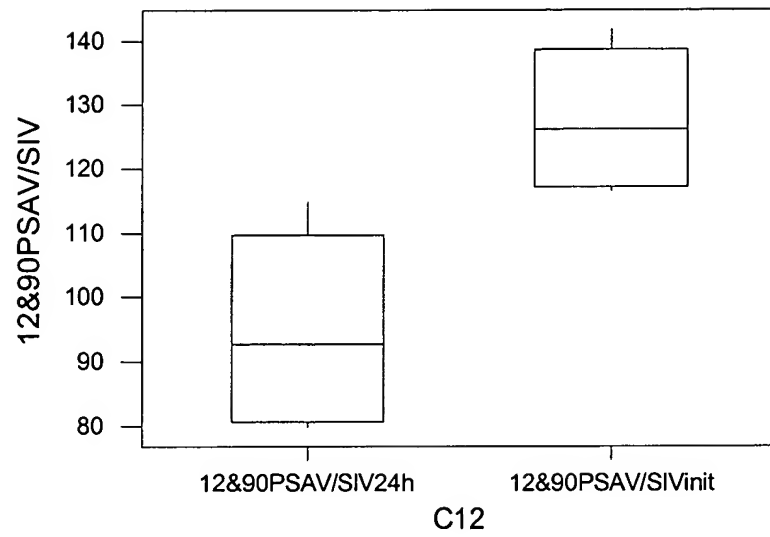
The graph below on PSA V shows a definite downtrend of peel strength values.



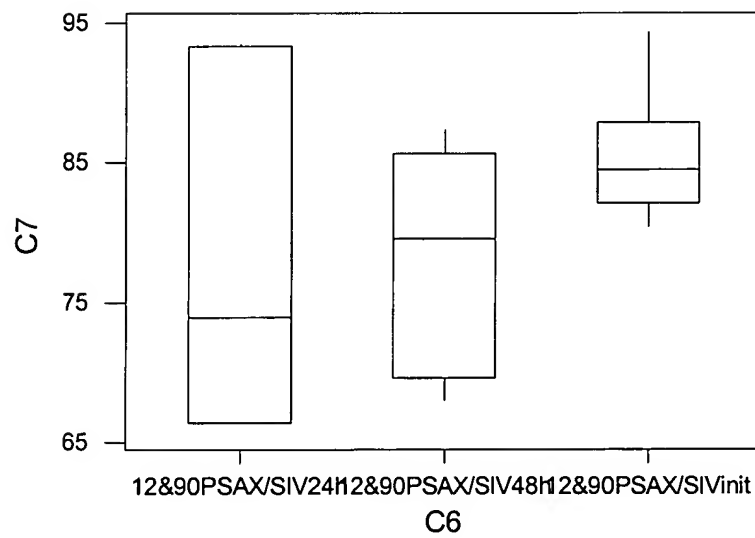
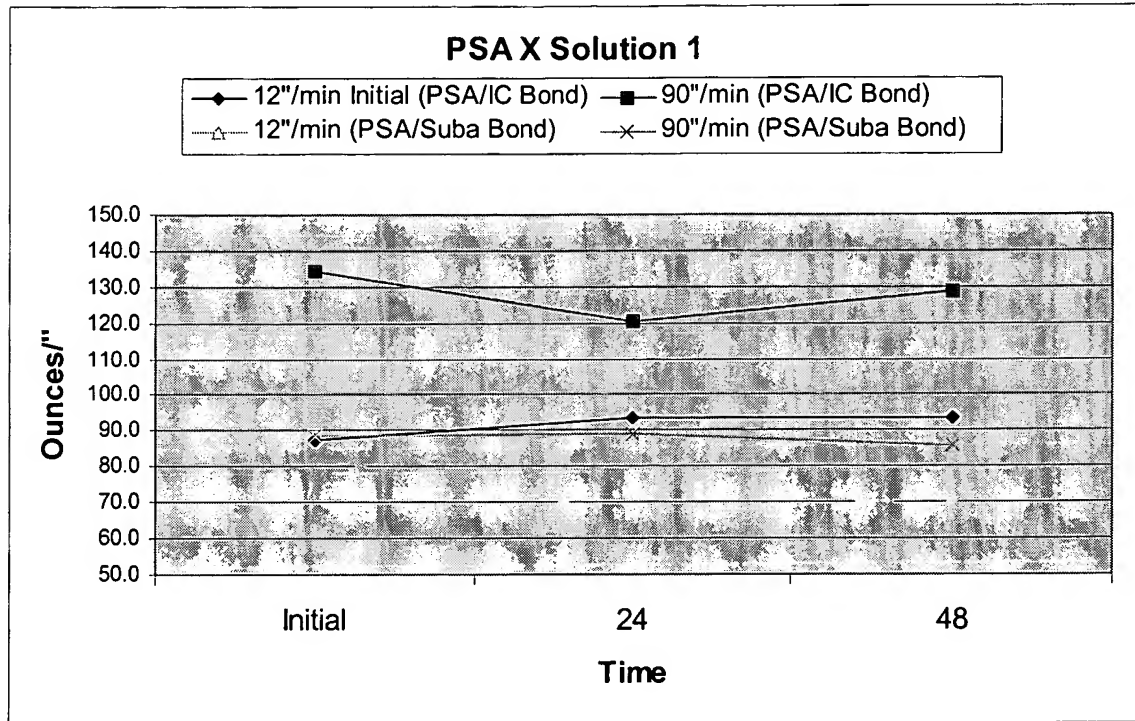
The graph below shows PSAV to IC is definitely different after immersion when comparing both test speeds at initial and after 24 hours(25 oz/in drop):

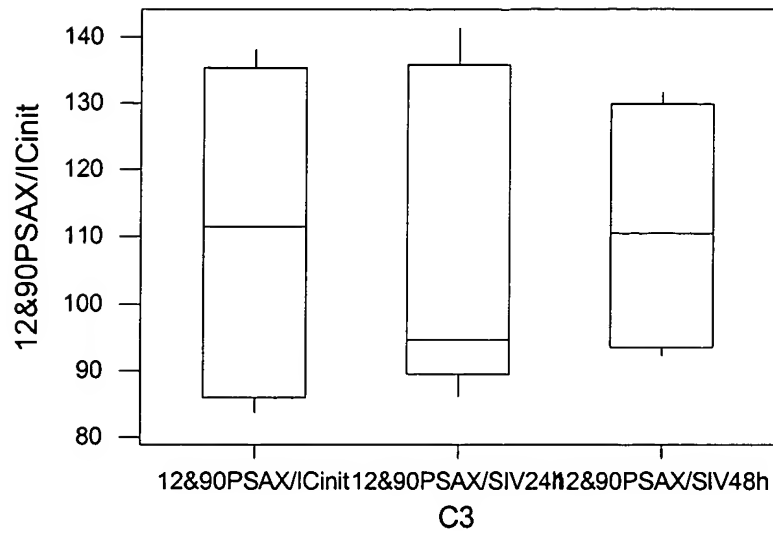


The PSA V bond to Suba is also affected by product immersion as shown below (35 oz/in drop):

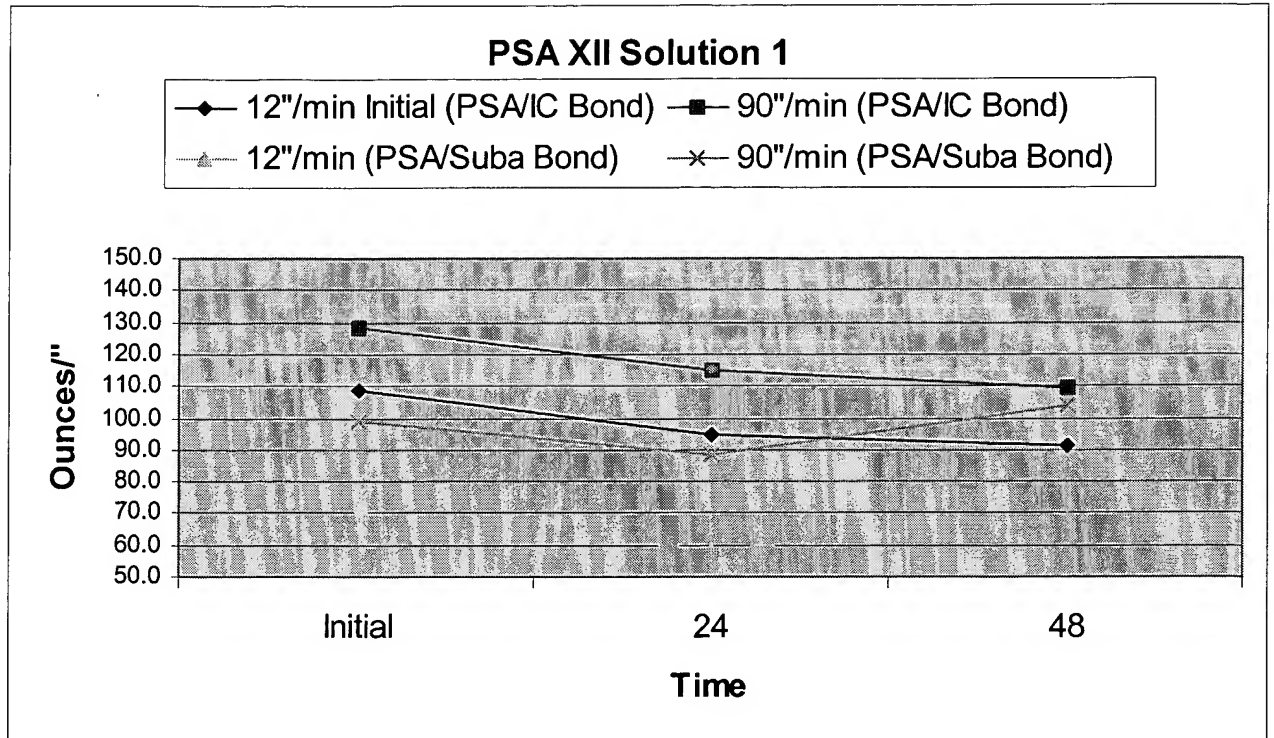


The graph below shows the PSAX is not affected on the PSA to IC bond.

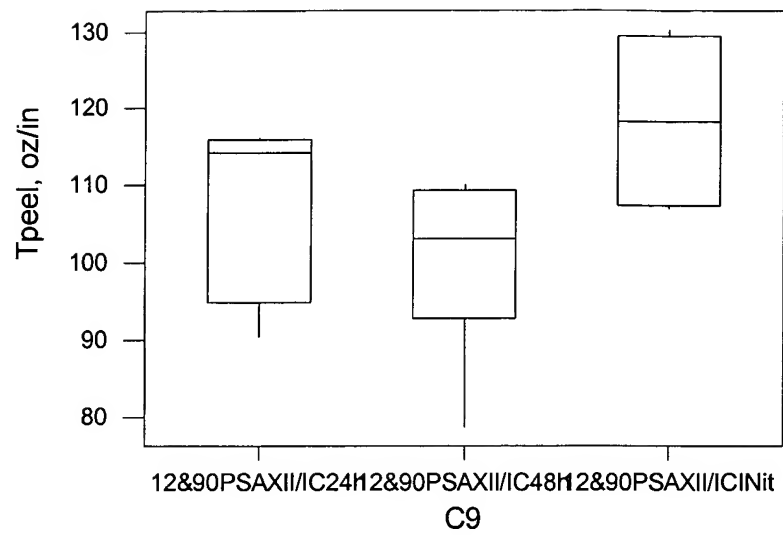




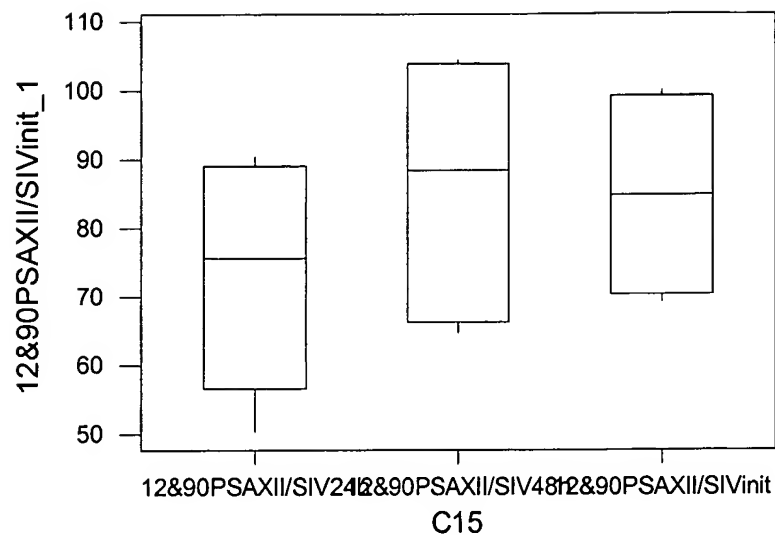
This graph shows that the PSA /Suba bond is the weakest bond with some reduction in peel values after the immersion.



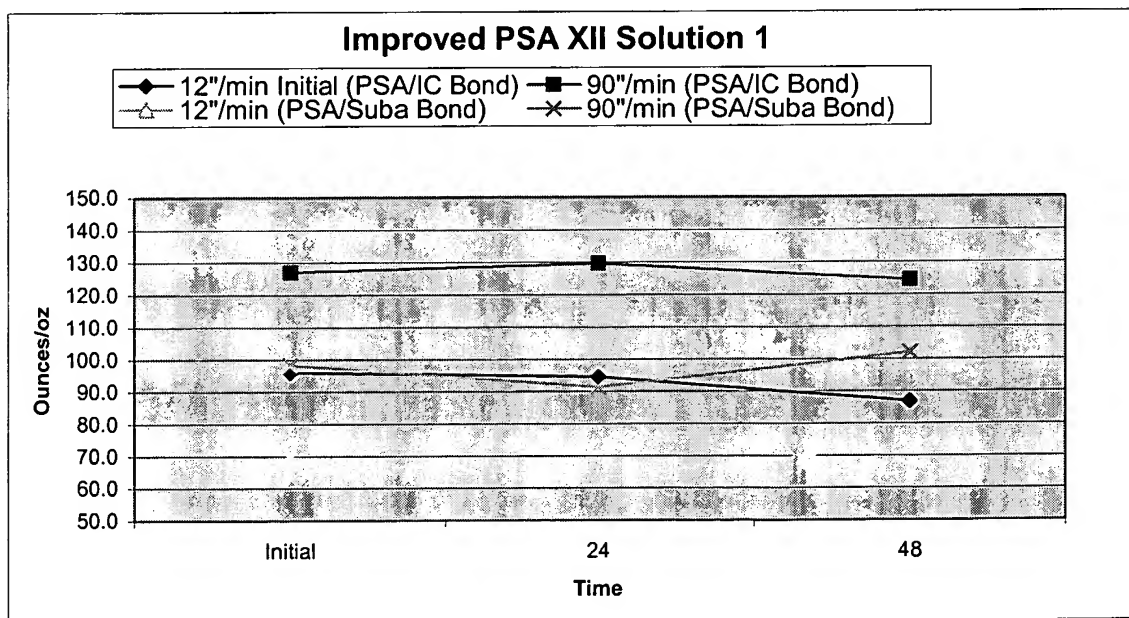
The graph below also shows that PSAXII peels to IC are statistically lower after 24 and 48 hours (15oz/in drop):



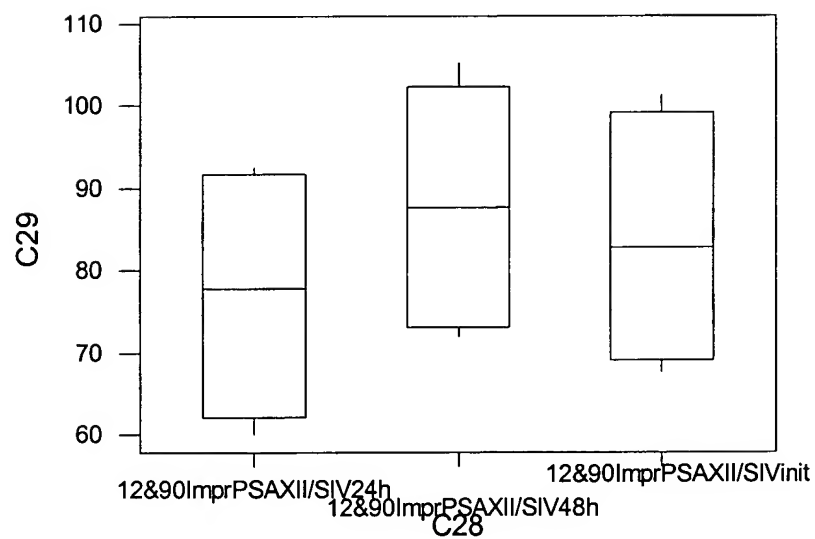
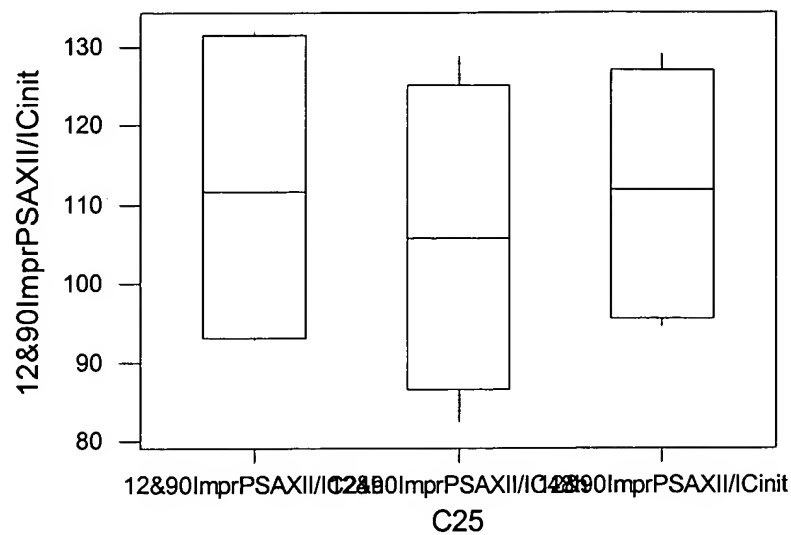
PSAXII bond to SIV was not significantly affected after 24 hours



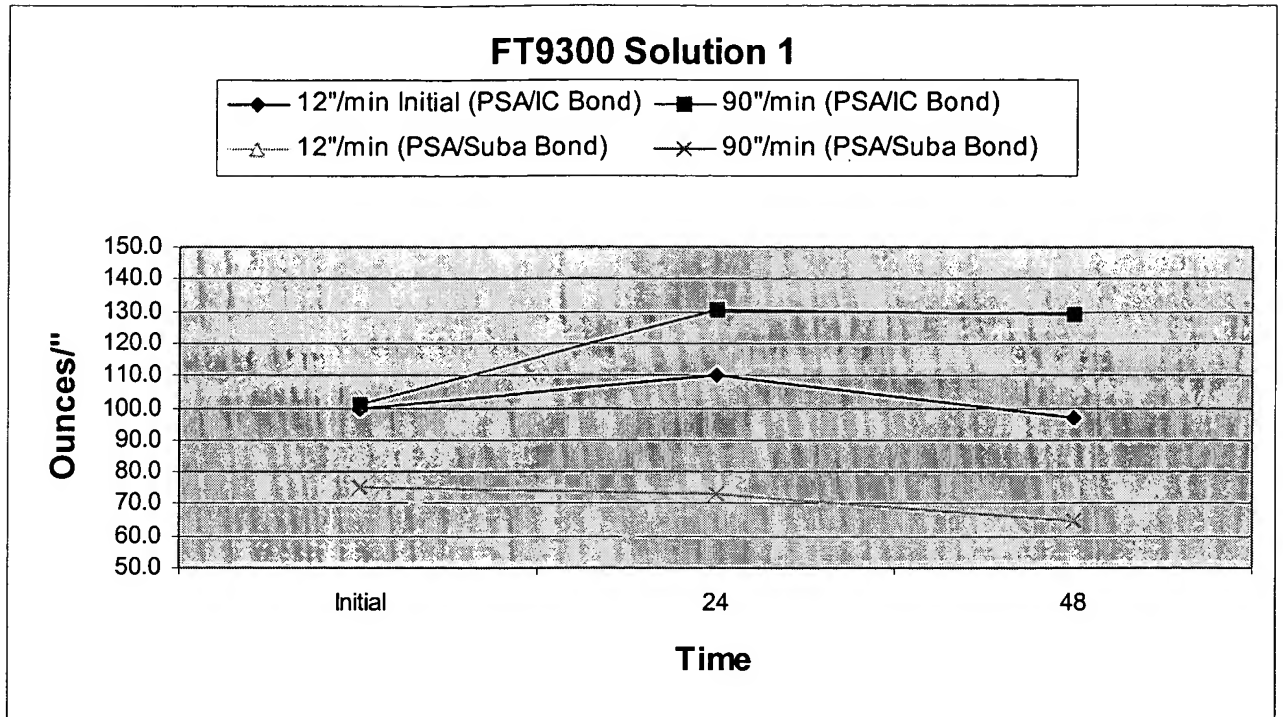
The improved PSAXII (lower residual cross link inhibitor levels shows less trend in lower peel values after immersion (except at 12"/min). This appears to reinforce our previous theory that indicated that the PSA XII cross linking mechanism was not previously well controlled during manufacturing.



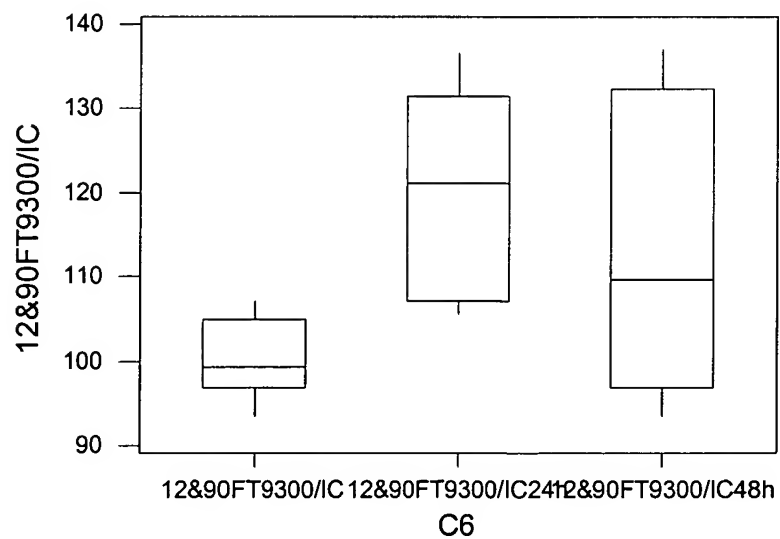
The improved PSAXII to IC and SIV bonds shows no downtrend after 24 and 48 hours:



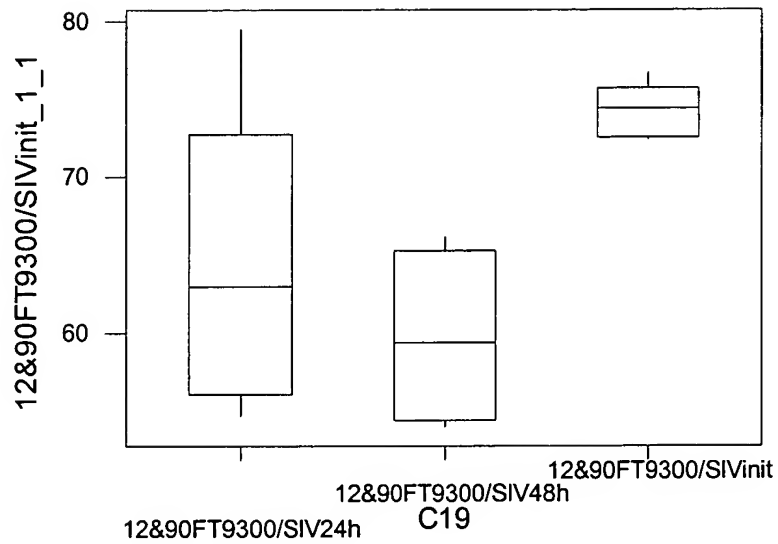
The FT9300 shows a downtrend in peel values after immersion at 12"/min



The graph below shows that the FT9300 bond to IC is not affected by the immersion

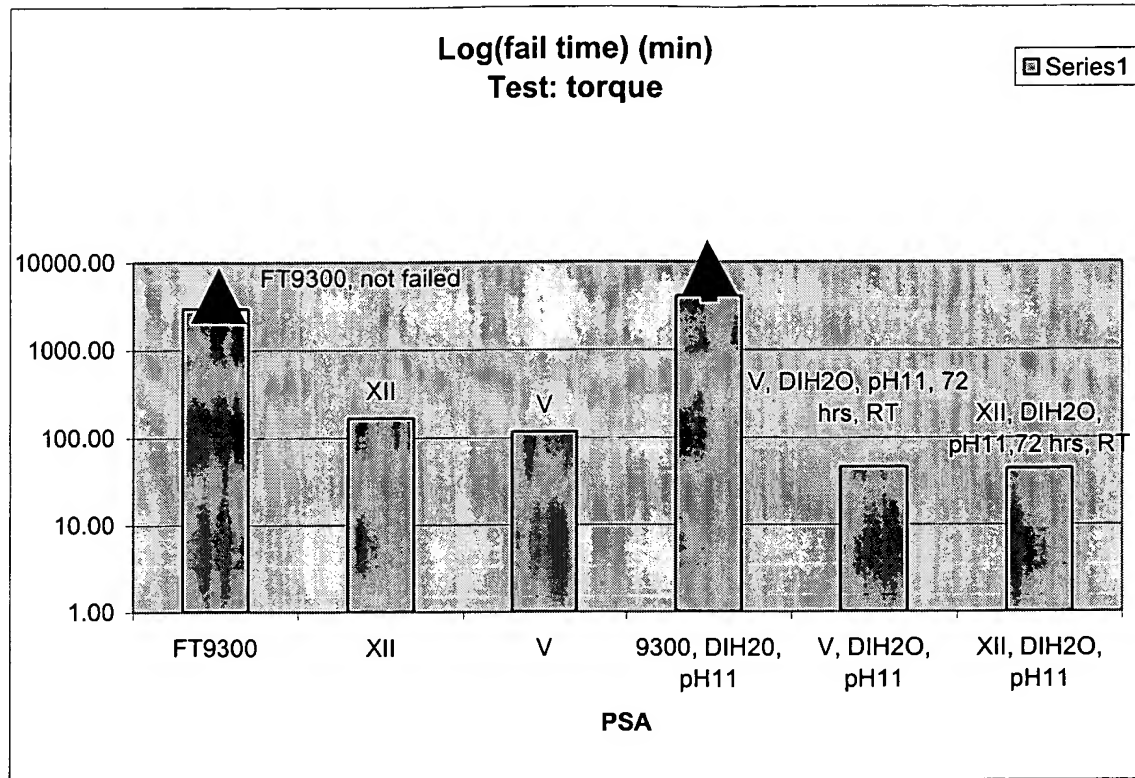


However, the bond to SIV was lower after 24 and 48 hours



II.4.1.2. Torque test data

This test setup produces shear forces on the interface. The graph below shows the torque test values after immersion.



This test shows that the FT9300 is not affected by the pH 11 solution after 72 hours. Whereas PSA V and XII are experiencing a drop in failure times.

Conclusions:

- For all PSA studied, the PSA/Suba interface represents the weakest peel adhesion values when compared to the PSA/IC interface. The trends appear more easily distinguished at 12"/min. PSA V exhibits the greatest downtrend in peel values to Suba after immersion.
- PSA V and PSAXII adhesion to IC is affected by immersion in solution 1 whereas FT9300 adhesion to IC was not reduced after slurry immersion.
- PSA V, XII, FT9300 Tpeel adhesion to SIV were affected after slurry immersion.
- The improved PSAXII (higher temperature drying to lower residual cross link inhibitor levels) peel adhesion to IC and SIV were no significantly different than initial Tpeels after 24 and 48 hours immersions.
- PSAX peel values to IC and SIV were not affected by immersion
- PSA V and PSAXII failure times were affected by the pH 11 solution whereas FT9300 failure time was not significantly affected by immersion in this solution.

II.5

III

Table of Contents

- I. Introduction
- II. Explore Chemical Spaces
- III. Explore Process Spaces
- IV. Non-Polishing Experiments
- V. Substitution of Key Ingredients
- VI. Additional Ingredients – “Helpers”
- VII. Investigation of Other, More Forward-Looking Ideas to Provide the Foundation for the Future of the Program

APPENDIX C1



Laurent S.
Vesier/NAmerica/Rodel
06/29/2005 01:07 PM

To Edwin Oh/NAmerica/Rodel@ROH
cc
bcc
Subject Fw: Adhesives

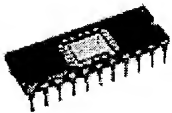
I have attached a project outline for adhesives improvement. It clearly shows our 9/26 date for review of hot melt vendors.

Thanks

Laurent Vesier | CMP Technologies | Rohm and Haas Electronic Materials | 451 Bellevue Road | Newark, DE 19713 |

Office: 302 366 0500x6314 Cell: 302-420 5166 | Fax: 302-453 1302 | www.electronicmaterials.rohmhaas.com

----- Forwarded by Laurent S. Vesier/NAmerica/Rodel on 06/29/2005 11:41 AM -----



Laurent S.
Vesier/NAmerica/Rodel
09/26/2002 09:30 AM

To Peter Freeman/NAmerica/Rodel, Colin Cameron
cc
Subject Adhesives

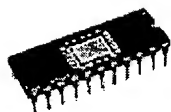
Peter, Colin

I have attached below tasks that we have ongoing for the adhesive project. Please let me know if you have questions. We have a weekly tuesday 2:30 meeting where we review updates so you are welcome to attend this meeting so you can give input on project direction.



Interfaceproject.mpx

APPENDIX C2



Laurent S.
Vesier/NAmerica/Rodel
06/29/2005 01:08 PM

To Edwin Oh/NAmerica/Rodel@ROH
cc
bcc
Subject

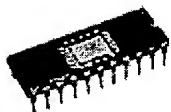
I am forwarding an email I had sent to John Roberts on a reactive hot melt vendor.

Thanks

Laurent Vesier | CMP Technologies | Rohm and Haas Electronic Materials | 451 Bellevue Road | Newark, DE 19713 |

Office: 302 366 0500x6314 Cell: 302-420 5166 | Fax: 302-453 1302 | www.electronicmaterials.rohmhaas.com

----- Forwarded by Laurent S. Vesier/NAmerica/Rodel on 06/29/2005 11:32 AM -----



Laurent S.
Vesier/NAmerica/Rodel
09/26/2002 03:05 PM

To Roberts John
cc
Subject

John

Cytec does not have Hot melts, but what do you think about a water based polyurethane system to try out?



I have attached a product bulletin ce-1175onecompPU.pc

APPENDIX C3



Laurent S.
Vesier/NAmerica/Rodel
06/29/2005 01:07 PM

To Edwin Oh/NAmerica/Rodel@ROH
cc
bcc
Subject Fw: Instron

I have attached an email of the test procedure we used for shear strength testing- a procedure we had to use for testing reactive hot melt adhesives.
Thanks

Laurent Vesier | CMP Technologies | Rohm and Haas Electronic Materials | 451 Bellevue Road | Newark, DE 19713 |

Office: 302 366 0500x6314 Cell: 302-420 5166 | Fax: 302-453 1302 | www.electronicmaterials.rohmhaas.com

----- Forwarded by Laurent S. Vesier/NAmerica/Rodel on 06/29/2005 11:39 AM -----



Laurent S.
Vesier/NAmerica/Rodel
10/02/2002 03:05 PM

To George Lamborn/NAmerica/Rodel, Mike Riello
cc Mark Boldizar/NAmerica/Rodel@EM
Subject Instron

Mike/George

I have attached a draft procedure for the instron from yesterday.

please let me know if you have any comments. For the immersion tests, I will send you the MSDS and appropriate safety steps in handling (ie PPE, etc) as well as show you where we are currently doing the samples immersions.

Thanks for your active training participation yesterday

NB: we don't need to put this procedure in Master control at this time as this is not a process control type measurement



Dynamic shear strength test procedure

APPENDIX C4



Laurent S.
Vesier/NAmerica/Rodel
06/29/2005 01:07 PM

To Edwin Oh/NAmerica/Rodel@ROH
cc
bcc
Subject Fw: Hot melt adhesives

I am forwarding an email sent to our Rohm and Haas research experts on Reactive Hot melts.
Thanks

----- Forwarded by Laurent S. Vesier/NAmerica/Rodel on 06/29/2005 11:36 AM -----



Laurent S.
Vesier/NAmerica/Rodel
10/03/2002 02:32 PM

To Rob Byrd/NAR/RohmHaas, Steven D Dr
Fields/NAR/RohmHaas
cc J. Costanzo/NAR/RohmHaas@ROH
Subject Hot melt adhesives

Hello,

Our Rodel, Newark, DE facility has a team looking into our purchased adhesives to reduce delamination at our customers.

In discussion with Jim Costanzo earlier, he indicated that you and others versed in specialty adhesives may be able to help us in selecting hot melt adhesives that could work well in our pads.

Our polyurethane polishing top pads are laminated to polyester fiber bottom pads and we are looking at direct coating the top pad with hot melt adhesives (PURs would be compatible to our polyurethane pads), then laminating to the bottom pad. We currently purchase PSAs for laminating the bottom to the top pad.

Would we be able to discuss this application with you and Jim within the next week or so?

We could travel to Springhouse to discuss.

APPENDIX D

John Roberts/NAmerica/Rodel
02/20/2003 01:48 PM

To Barbara Wiley/NAmerica/Rodel@EM
cc
bcc
Subject Re: Hot melt adhesive NOI

02029

Barb, could you add Larent Vesier's name to this application? He has provided substantial contribution on this, and it was my failing to have his name added in the first place.

John Roberts
302-366-0500 x6281
Cell 302-420-7088

Barbara Wiley/NAmerica/Rodel

Barbara
Wiley/NAmerica/Rodel
10/09/2002 03:42 PM

John Roberts/NAmerica/Rodel@EM, Ken
Benson/NAmerica/Rodel@EM, David
To James/NAmerica/Rodel@EM, Cathie
Markham/NAmerica/Rodel@EM, Gerry
Kita/NAmerica/Rodel@EM

cc

Subject Hot melt adhesive NOI

This is NOI 02029.

David and Cathie: Please review for discussion at the IP meeting on 10/14.

Thanks,

Barb

----- Forwarded by Barbara Wiley/NAmerica/Rodel on 10/09/2002 03:35 PM -----

Ken Benson
10/09/2002 02:52 PM

To: Barbara Wiley/NAmerica/Rodel@EM
cc: Gerry Kita/NAmerica/Rodel@EM, David
James/NAmerica/Rodel@EM
Subject: Hot melt adhesive NOI

Barb, Please give this a number and we will discuss it at our next pad meeting. Thanks, Ken

----- Forwarded by Ken Benson/NAmerica/Rodel on 10/09/2002 02:49 PM -----

John Roberts
10/09/2002 12:50 PM

To: Ken Benson/NAmerica/Rodel@EM, David
James/NAmerica/Rodel@EM
cc:
Subject: Hot melt adhesive NOI



HMA NOI.doc

John Roberts
302-366-0500 x6281
Cell 302-420-7088